



PATENT SPECIFICATION

686,876

Date of Application and filing Complete Specification: Jan. 15, 1951.

No. 1048/51.

Application made in France on Jan. 19, 1950.

Complete Specification Published: Feb. 4, 1953.

Index at acceptance:—Class 1(ii), A13.

COMPLETE SPECIFICATION

Method of Manufacture of Cristobalite

We, PROSILIS, of 8, rue d'Anjou, Paris, France, a Body Corporate organized under French law, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to a method of manufacture of cristobalite.

Cristobalite is a crystalline variety of silica, the stability range of which is between 1470° C. and 1710° C. (melting point of the said cristobalite).

The advantage of cristobalite over vitreous silica, in their use as refractories, is that cristobalite does not subside under loads at high temperature, like vitreous silica, which fact allows its use as refractory up to 1700° C. Moreover, the crystals formed are microscopic, and it is easy to obtain very fine powders of practically pure silica.

There is already known a method of manufacture of cristobalite, by calcination of ground quartz having a well-defined granular size.

There has already been proposed (see Patent Specification 485,871) in the manufacture of shaped articles of silica, a method of increasing their heat stability which consists in subjecting comminuted quartzite to heat in admixture with a granular agent for promoting the allotropic change from quartzite to tridymite and/or cristobalite, said promoting agent including one or more of the halides or silicofluorides of the alkali and/or alkaline earth metals, to convert it into cristobalite and/or tridymite. In the said Specification, it is recalled that the heating of ground quartzite with milk of lime cements the grains of quartzite together, thereby imparting strength to the bricks produced, and also that this heating operation has the very important effect of changing the quartzite crystals to one of

the allotropic forms of silica, such as tridymite or cristobalite; to obtain the cristobalite form, it is necessary to heat the material to about 1500° C.

There have also been published the results of laboratory experiments on the conversion of quartz into tridymite and cristobalite, but these were only investigations in the laboratory and there was no question of a complete conversion into cristobalite.

The present invention consists of an industrial method of manufacture of pure cristobalite in powder form, characterized in that very pure siliceous sand or other pure siliceous material, assaying at least 99.8% of silica, is melted with a small proportion of catalyst equal to or less than 0.1% consisting of an oxide of an alkali or alkaline earth metal, or an equivalent proportion of a salt of such metal capable of conversion into oxide, and that the molten mass obtained is cooled slowly.

There is thus obtained a friable mass, giving a white powder, which is pure cristobalite. The quantity of catalyst to be used depends upon the activity thereof; the alkali and alkaline earth metals can be classified in the following order of decreasing activity of their oxides or salts:—Li, Na, ... Ca, Be, ... and the activity must be great enough for the molten mass to crystallize completely on cooling.

By way of example, and without this limiting the invention in any way, the following mode of operation may be followed:

Fontainebleau sand, having added thereto 0.1% by weight of sodium oxide, introduced in the form of a solution of sodium carbonate, is packed around a graphite electrode.

The mixture is melted by passing a high current of about 1000 amperes at 40 volts through the electrode. Upon subse-

quent slow cooling, the mass obtained gives a fissured block of cristobalite, which breaks up easily and is converted into a fine powder which can be employed for the construction of refractory puddle-work, cement for the pouring of metals, fillers and articles of any kind according to the ceramic art.

What we claim is:—

- 10 1. An industrial method of manufacture of pure cristobalite, in powder form, characterized in that very pure siliceous sand or other pure siliceous material, assaying at least 99.8% of silica, is melted with a small proportion of catalyst
15 equal to or less than 0.1% consisting of an oxide of an alkali or alkaline earth metal, or an equivalent proportion of a salt of

such metal capable of conversion into oxide, and that the molten mass obtained is cooled slowly.

2. The application of cristobalite obtained by the method according to claim 1, as base for manufacture of refractory cements, articles or puddle-work.

3. Refractory cements, articles or puddle-work having a base of cristobalite obtained by the method according to claim 1.

Dated the 15th day of January, 1951.

For the Applicants,
FEENY & FEENY,
Chartered Patent Agents,
2a, Charlwood Place,
Westminster, S.W.1.

Leamington Spa: Printed for Her Majesty's Stationery Office, by the Courier Press.—1953
Published at The Patent Office, 25, Southampton Buildings, London, W.C.2. from which
copies may be obtained.